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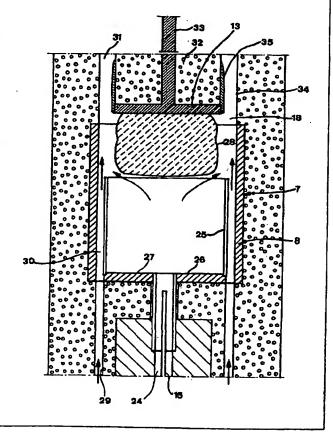
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(54) Title: A DEVICE FOR EPITAXIALLY GROWING OBJECTS AND METHOD FOR SUCH A GROWTH

#### (57) Abstract

A device for epitaxially growing objects of for instance SiC by Chemical Vapour Deposition on a substrate (13) has means (15) for feeding a first flow of at least a carrier gas and reactive gases needed for the growth into a susceptor (7) towards the substrate and means (25, 29) adapted to conduct an additional flow of gases not participating in said growth substantially in parallel with said first flow along circumferential walls (8) of the susceptor past the substrate and out through outlet means (31) of the susceptor. The device comprises means (25) arranged to separate the path of said first flow from said circumferential walls and guide it at a distance therefrom to the region of said object (28).



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A device for epitaxially growing objects and method for such a growth

#### TECHNICAL FIELD OF THE INVENTION AND PRIOR ART

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The present invention relates to a device for epitaxially growing objects of one of a) SiC, b) a Group III-nitride and c) alloys thereof by Chemical Vapour Deposition on a substrate comprising a susceptor having circumferential walls surrounding a room for receiving the substrate, means for feeding a first flow of at least a carrier gas and reactive gases needed for the growth into the susceptor towards the substrate, outlet means located behind the substrate for letting a second flow of gases out of the susceptor and means for heating said circumferential walls and by that the substrate and said reactive gases above a temperature level from which the reactive gases start to decompose into substances depositing on the substrate for the growth of a said object, said feeding means comprising a conduit emerging into said room and directing said first flow towards the substrate, as well as a method for epitaxially growing such objects according to the preamble of the appended independent method claim.

Accordingly, the invention is applicable to growth of SiC, Group III-nitrides and alloys thereof, but the common problem when such objects are to be grown of a high crystalline quality and at a reasonable growth rate from the commercial point of view, i.e. at a relatively high flow rate of said reactive gases and at a comparatively high temperature, while forming objects having a considerable size in one

growth run will now by way of a non-limitative example be further explained for SiC.

When SiC single crystals are grown at such a reasonable growth rate the rate of reactive gases, mostly silane and propane, decomposed is rather high, and the substances, accordingly silicon and carbon, resulting from said decomposition will not only be deposited on the object grown but also on the walls of the outlet means. A complete growth run may have a duration of several tenths of hours if large crystals are to be grown, but it may happen that the outlet means are completely blocked already after only half an hour of growth.

Thus, the uniformity of gas flow will first be destorted as 15 long as the cross-section of the free passage of the outlet means is gradually reduced, and when the outlet means is completely blocked, the gas pressure inside said room of the susceptor will increase, since the gases introduced into said room have to escape therefrom through said outlet 20 means, until the pressure is that high that the carrier of the substrate and the substrate will be forced away from said room, so that the substrate and the object grown will be located obliquely to the direction of said first gas flow. Thus, the quality of the object grown in that way will 25 then be dramatically reduced, so that the growth has in practise to be terminated much earlier than desired. A device of this type is described in the U.S. patent application 08/511 324 (corresponding to the Swedish patent application 9502288-5). 30

### SUMMARY OF THE INVENTION

The object of the present invention is to advise a solution to the problem discussed above by providing a device and a method making it possible to epitaxially grow objects of

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SiC, a Group III-nitride or alloys thereof at a high growth rate while still obtaining a high crystalline quality of the object grown and be able to carry out the growth until an object of the size desired has been grown.

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This object is in accordance with the invention obtained by providing a device defined in the introduction with means adapted to conduct an additional flow of gases not participating in said growth substantially in parallel with said first flow along said circumferential walls past the substrate and out through said outlet means for preventing said substances deriving from reactive gases decomposed from moving along said circumferential walls past the substrate and the object grown thereon and keeping at least a wall of said outlet means substantially free from such substances and instead forcing them towards said object, and means arranged to separate the path of said first flow from said circumferential walls and guide it at a distance therefrom to the region of said object, so that the first flow will be separated from said additional flow to this region. Since a first gas flow containing the reactive gases will in this way meet said additional gas flow in the region of the object grown, and the additional gas flow will travel along said circumferential walls delimiting said room of the susceptor, this additional flow will keep the depositions of substances resulting from the decomposition of the reactive gases to the object side and spread them out over some distance to prevent blocking, so that it will keep a clean path at all times for gases to leave the susceptor through the outlet means. Thus, the depositions are forced to be made on the object grown instead of on walls defining the outlet means.

According to a preferred embodiment of the invention said separating means comprises second circumferential walls extending in said room substantially in parallel with the cir-

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cumferential walls of said room to said region for conducting said additional gas flow therebetween and said first flow within the limitation of said circumferential walls. In this way said additional gas flow will be kept in a path along said circumferential walls defining said room and separated from said first gas flow in a simple way so as to efficiently block the way of substances deriving from reactive gases decomposed to said circumferential walls when the flows meets beyond the end of said second circumferential walls.

According to another preferred embodiment of the invention said separating means ends before the intended location of an upstream end of an object grown as seen in the direction of said flows. This is an advantageous feature, since the additional gas flow will then meet the gases coming from said first flow so early that it may efficiently influence said substances to be deposited on such parts of the object grown where the growth is intended to take place without any risk of blocking of said outlet means.

According to another preferred embodiment of the invention the distance between the separating means and the circumferential walls of the susceptor is smaller than the width of the free passage provided between the object grown and the circumferential walls of the susceptor, which is preferable for preventing an intermixing of said additional gas flow with the gases deriving from the first gas flow.

According to another preferred embodiment of the invention said conduit arranged to conduct said first flow emerges into the susceptor at a first end of said room and the substrate and the outlet means are located at an opposite second end of said room, said circumferential walls of the susceptor extending from said first to said second end, said conduit for the first flow emerges into said room substan-

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tially centrally at said first end and a circumferential ring-like second conduit emerges into said room at the circumferential walls of the susceptor at said first end and is adapted to conduct said additional gas flow. Said additional gas flow may in this way be fed to the susceptor and given the direction desired along said circumferential walls delimiting the susceptor room for efficiently prevent depositions of substances deriving from reactive gases decomposed on said circumferential walls or walls of said outlet means in prolongation thereof.

According to another preferred embodiment of the invention said means for conducting said additional flow is arranged to conduct a flow of an inert gas. The choice of an inert gas for said additional gas flow is advantageous since this gas may not be deposited on the object grown or disturb said growth by reaction with walls or other gas species or be deposited on walls of the outlet means.

According to another preferred embodiment of the invention 20 said means for conducting said additional flow is arranged to conduct a flow of a gas having a high thermal conductivity. Thanks to this fact heat will efficiently be transferred from said circumferential walls through said addiseparating means for heating tional gas flow to said 25 thereof and by that for heating said first gas flow, so that the temperature thereof will be as high as desired and the thermal equilibrium favourable for said growth will be established in said room. When said circumferential walls are heated by induction caused by a Rf-field, it may in this way 30 also be possible to use a material not coupling to the Rffield for said separating means. For the sake of heat transfer to said first gas flow it would be advantageous to make said separating means rather thin, so that they may be easily heated to a desired temperature level. It has turned 35 out that He is very well suited to be used as the gas for

said additional flow, since it is inert and has a high thermal conductivity.

According to another preferred embodiment of the invention said device comprises a carrier for the substrate, and this carrier and by that the substrate and the object are arranged displacable in the opposite direction to the growth direction of said object. This means that even if there will be some growth on the sides of the object grown as seen in the direction of said gas flows, this will not constitute any problem, since a displacement of said carrier and by that the object in a direction opposite to the growth direction of said object at suitable time intervals means that the outlet will never be blocked provided that the cross-section thereof is large enough from the beginning.

The corresponding characteristics and advantages thereof are found in a method for epitaxially growing objects according to the invention defined in the appended method claims.

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Further preferred features and advantages of the device and method according to the invention will appear from the following description and the other dependent claims.

#### 25 BRIEF DESCRIPTION OF THE DRAWINGS

With reference to the appended drawings below follows a specific description of preferred embodiments of the invention cited as examples.

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In the drawings:

Fig. 1 is a longitudinal cross-section view of a prior art device of the type described in the U.S. patent ap-

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plication 08/511 324 for the growth of objects by Chemical Vapour Deposition, and

Fig. 2 is an enlarged cross-section view of a susceptor and the gas inlet and outlet parts thereof in a device of the type shown in Fig. 1 according to a preferred embodiment of the invention.

# DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT OF THE INVENTION

Fig. 1 shows schematically a device of the type described in the US patent application 08/511 324, but it will still be described here, since the device according to the invention may have the same principle construction as that device and only differ therefrom by the particular characteristics defined in the present invention. This device is suited for epitaxially growing SiC by Chemical Vapour Deposition on a SiC substrate. The device is shown in a simplified manner, and it is obvious that the device in question also comprises other means, such as pumps, but conventional equipment having nothing to do with the inventional problem has been omitted for the sake of clearness and concentration to the invention.

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This device comprises a casing 1 constituted by a tube 2 of quartz extending substantially vertically and two opposite end flanges 3 and 4. The end flange 4 is preferably removable so as to get access to the interior of the tube 2. A conduit 5 for supplying a flow of a carrier gas and reactive gases, preferably silane and propane, for said growth to the substrate is introduced through the lower end flange 3. The conduit 5 is connected to separate conduits 20-23 leading to sources for different gases for said growth, such as silane and propane, and said carrier gas, and these conduits are provided with flow regulating means not shown for regulating

the content of each component in the gas flow in the conduit 5. The conduits 20-23 are in Fig. 1 for the sake of clearness illustrated as emerging into the conduit 5 close to the casing 1, but in practise they will probably be at a greater distance therefrom.

Furthermore, the device comprises a funnel 6 for concentrating the gas flow from the conduit 5 into a susceptor 7. The susceptor 7 shown in Fig. 1 is adapted for epitaxially growing layers of SiC. The susceptor is substantially cylindrical with circumferential walls 8 of a substantial uniform thickness. The walls are made of graphite, but they are internally coated by a layer of SiC 9 or alternately covered by a cylindrical plate made of SiC. The space surrounding the susceptor is enclosed and filled by graphite foam 10 for thermal insulation for protecting the surrounding quartz tube 2. Rf-field radiating means 11 in form of a Rf-coil surrounds the tube 2 along the longitudinal extension of the susceptor 7. This heating means 11 is arranged to radiate a Rf-field uniformly heating the walls 8 of the susceptor and thereby the gas mixture introduced into the susceptor.

The susceptor 7 comprises a lid 12 of the same material as the rest of the susceptor, on the lower side of which a SiC substrate 13 is arranged and which may be removed from the rest of the susceptor so as to remove the substrate after a layer has been grown thereon. The lid 12 is provided with peripheral gas outlet holes 14, so that a preferably laminar gas flow will enter the susceptor room 18 through a lower inlet conduit 15 and flow close to the substrate and leave the susceptor through the upper outlets 14 and then the device through a conduit 16 optionally connected to a pump not shown.

The temperature inside the susceptor 7 may be checked pyrometrically through looking into the susceptor 7 through a window indicated at 17.

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In such a device the heating means 11 will heat the suscep-5 tor walls 8 and by that the susceptor room 18 and the substrate 13 and the gases introduced into the susceptor room through the conduit 15, so that the reactive gases will decompose and deposit on the substrate for the growth. When high growth rates are desired it is necessary to raise the 10 temperature inside the susceptor to a high level, such as for instance above 2000°C and it may not be avoided that the temperature in the conduit 15 will be that high that reactive gases, especially silane, will be decomposed already in this conduit and deposited on the inner wall of that 15 conduit, which will result in problems with plugging of the inlet; but this problem is solved by arranging a third conduit 24 substantially centrally inside the inlet conduit 15 (see Fig. 2) for conducting substantially the entire flow of reactive gases and making the space of the first conduit 20 15 surrounding said third conduit to conduct the flow of a carrier gas and terminate said third conduit 24 at a distance from said room, so that the reactive gases will not be decomposed in said second conduit and if they decomposed in said first conduit before entering said room 25 they will not reach the walls of said first conduit for These features are new within the depositing thereon. technique but are an object of another patent application filed for that invention and will not be described more in detail here. 30

In a device of the type shown in Fig. 1 parasitic depositions of primarily silicon and carbon will take place in the outlet holes 14, so that there is a risk that these holes are completely blocked and the growth has to be interrupted

a long time before the SiC-crystal has been grown to the size desired.

It shall now be explained with reference to Fig. 2 how the present invention solves this problem with blocking of the 5 outlet holes of a device of this type. A cylinder 25, preferably made of SiC and having thin second circumferential walls and a smaller outer diameter than the inner diameter of the susceptor, is arranged substantially concentrically to the circumferential walls 8 of the susceptor at a first 10 gas inlet end 26 of the susceptor. The cylinder 25 is arranged on the end member 27, with the present orientation of the device the bottom, so that the first gas flow emerging into the room of the susceptor through the conduit 15 will move towards the object 28 grown on the substrate 13 without 15 any possibility to reach the circumferential walls 8 of the susceptor.

The device further comprises a circumferential ring-like second conduit 29 emerging into the room of the susceptor at 20 the circumferential walls of the susceptor at said first end 26 thereof for making it possible to feed a gas flow to the circumferential space 30 formed between said second circumferential walls of the cylinder 25 and the circumferential walls 8 of the susceptor. Furthermore, the outlet means com-25 prises a circumferential opening 31 surrounding said substrate 13 and a carrier 32 for the substrate, so that the gases may pass an object 28 grown, the substrate 13 and said carrier 32 and leave said room 18 of the susceptor. The carrier for the substrate may be displaced in the direction 30 away from said first end 26, preferably by having an axle 33 thereof received in threads so that the carrier may be gradually screwed out of the susceptor as the thickness of the object grown increases. This is done so that the cylinder 25 will with respect to the direction of the gas flows 35 end before the upstream end of said object 28, and it may be mentioned that it has in a particular case turned out to be advantageous to have the cylinder 25 ending approximately 5 mm from said upstream end of the object when an object having a diameter of approximately 70 mm is grown.

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The function of the device with respect to those parts described with reference to Fig. 2 is as follows. A first gas flow containing a carrier gas and reactive gases needed for the growth, such as silane and propane in the case of growing an object of SiC, is introduced into the susceptor room 18 through the conduit 15 in the way described further above. An additional flow of gases not participating in said growth is at the same time introduced through the second conduit 29 in the space 30 between the inner cylinder 25 and the circumferential walls 8 of the susceptor and in this way brought to flow along said circumferential walls 8 of the susceptor. A gas being inert and having a high thermal conductivity is preferably used for said additional flow, and it has turned out that He is very well suited therefor. The first gas flow and said additional gas flow will meet in a region of the object 28, where the cylinder 25 ends. The gas from the additional gas flow will continue to flow along said circumferential walls 8 of the susceptor and out through the opening 31 along the outer walls 34 thereof and influence the gas streams, which besides a carrier gas, such as H2, contains substances deriving from said reactive gases decomposed, to travel at the object side of the free passage in question, so that this substances will tend to be deposited on the object, and perhaps may after considerable period of time also some depositions be made on the wall of the carrier as indicated at 35. Depending on the temperature gradients, the amount of said substances in the gas-phase and the depletion there will likely be some growth on the sides of the crystals 28, which if this is allowed to be continued for a long time, may cause blocking of the passage between the object 28 and the circumferential walls 8, but

out of the susceptor room as the thickness thereof increases during the growth. Thus, the effect of said additional gas, which may be called a "cleaning-gas", will be to keep the depositions to the object side and to spread them out over some distance to prevent blocking of the outlet of the susceptor. It is essential for the function of the device that the mixture between the additional gas flow and the gas flow deriving from said first gas flow is low, and it will therefore be preferred that the additional gas flow is laminar.

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The invention is of course not in any way restricted to the preferred embodiment of the device and method described above, but several possibilities to modifications thereof would be apparent for a man skilled in the art without departing from the basic idea of the invention.

As already mention, the invention is also applicable to growth of a Group III-nitride or an alloy of Group III-nitrides or an alloy of SiC and one or more Group III-nitrides, for which the corresponding positive result may be expected.

The definition "object" in the claims is made for including the epitaxial growth of all types of crystals, such as layers of different thicknesses as well as thick boules.

All definitions concerning the material of course also include inevitable impurities as well as intentional doping.

Although it has been spoken about diameters for the room of the susceptor and the separating means separating the first gas flow from the circumferential walls of the susceptor, this is not to be interpreted as a restriction to exactly circular cross-sections of these parts but other shapes may also be possible.

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"Second flow of gases" in the preamble of the independent patent claims is coming from said first flow, but it is defined as a second flow, since it may have another composition than the first flow due to decompositions of reactive gases therein during the transport through the susceptor.

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Although it has been indicated in the Figures that the flow of gases to the substrate is substantially vertical, it is within the scope of the invention to arrange the device with an arbitrary extension direction, such as for instance horizontal.

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#### **Claims**

1. A device for epitaxially growing objects of one of a) SiC, b) a Group III-nitride and c) alloys thereof by Chemi-5 cal Vapour Deposition on a substrate (13) comprising a susceptor (7) having circumferential walls (8) surrounding a room (18) for receiving the substrate, means (15, 24) for feeding a first flow of at least a carrier gas and reactive gases needed for the growth into the susceptor towards the 10 substrate, outlet means (31) located behind the substrate for letting a second flow of gases out of the susceptor and means (11) for heating said circumferential walls and by that the substrate and said reactive gases above a temperature level from which the reactive gases start to decompose 15 into substances depositing on the substrate for the growth of a said object, said feeding means comprising a conduit (15) emerging into said room and directing said first flow towards the substrate, characterized in that the device further comprises means (25, 29) adapted to conduct an addi-20 tional flow of gases not participating in said growth substantially in parallel with said first flow along said circumferential walls (8) past the substrate and out through said outlet means for preventing said substances deriving from reactive gases decomposed from moving along said cir-25 cumferential walls past the substrate and the object grown thereon and keeping at least a wall of said outlet means substantially free from such substances and instead forcing them towards said object, and that it further comprises means (25) arranged to separate the path of said first flow 30 from said circumferential walls and guide it at a distance therefrom to the region of said object, so that the first flow will be separated from said additional flow to this region.

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2. A device according to claim 1, <u>characterized</u> in that said separating means comprises second circumferential walls (25) extending in said room substantially in parallel with the circumferential walls (8) of said room (18) to said region for conducting said additional gas flow therebetween and said first flow within the limitation of said second circumferential walls.

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- 3. A device according to claim 1 or 2, <u>characterized</u> in that said separating means (25) ends before the intended location of an upstream end of an object (28) grown as seen in the direction of said flows.
- 4. A device according to any of claims 1-3, characterized in that the distance between the separating means (25) and said circumferential walls (8) of the susceptor (7) is smaller than the width of the free passage provided between the object grown and said circumferential walls of the susceptor.

5. A device according to claim 2, <u>characterized</u> in that said second circumferential walls are formed by an inner cylinder (25) substantially concentrically arranged in said room (18) with respect to the circumferential walls (8) of the susceptor.

- 6. A device according to claim 2, <u>characterized</u> in that said second circumferential walls (25) are made of SiC.
- 7. A device according to any of claims 1-6, characterized in that said conduit (15) arranged to conduct said first flow emerges into the susceptor at a first end (26) of said room and the substrate (13) and the outlet means (31) are located at the opposite second end of said room, said circumferential walls (8) of the susceptor extending from said first to said second end.

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- 8. A device according to claim 7, characterized in that said conduit (15) for the first flow emerges into said room substantially centrally at said first end (26), that a circumferential ring-like second conduit (29) emerges into said room at the circumferential walls of the susceptor at said first end and is adapted to conduct said additional gas flow.
- 9. A device according to any of claims 1-8, characterized in that said outlet means comprises a circumferential opening (31) surrounding said substrate (13) and a carrier (32) for the substrate, so that gases may pass an object grown, the substrate and said carrier and leave said room of the sustential ceptor.
  - 10. A device according to any of claims 1-9, characterized in that it comprises a carrier (32) for the substrate (13), and that this carrier and by that the substrate and the object are arranged displaceable in the opposite direction to the growth direction of said object.
  - 11. A device according to any of claims 1-10, characterized in that said means (25, 29) for conducting said additional flow is arranged to conduct a flow of an inert gas.
    - 12. A device according to any of claims 1-11, characterized in that said means (25, 29) for conducting said additional flow is arranged to conduct a flow of a gas having a high thermal conductivity.
    - 13. A device according to a any of claims 1-12, <u>characterized</u> in that said means (25, 29) for conducting said additional flow in the form of He.

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14. A device according to any of claims 1-13, characterized in that said means (25, 29) for conducting said additional flow is arranged to conduct a substantially laminar additional flow of gases.

15. A device according to any of claims 1-14, characterized in that it is adapted for growth of objects of SiC.

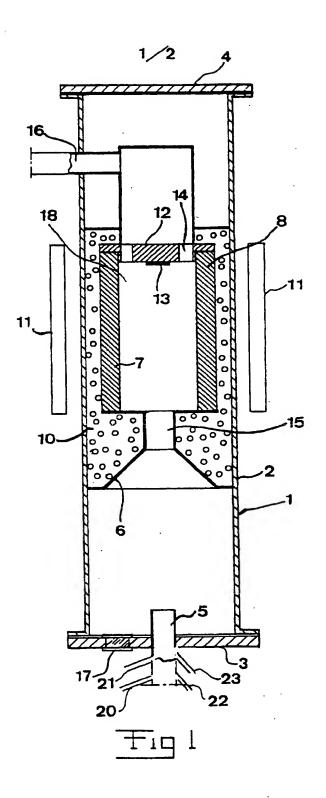
- 16. A device according to claim 15, <u>characterized</u> in that said heating means is arranged to heat said circumferential walls and by that the substrate and said reactive gases at a temperature above 1500°C.
- 17. A device according to claim 15 or 16, <u>characterized</u> in that said feeding means is arranged to feed silane and propane as reactive gases in said first flow.
- 18. A method for epitaxially growing objects of one of a) SiC, b) a Group III-nitride and c) alloys thereof by Chemical Vapour Deposition on a substrate (13) received in a room 20 (18) of a susceptor (7) having circumferential walls (8), in which a first flow of at least a carrier gas and reactive gases needed for the growth is fed into the susceptor towards the substrate, a second flow of gases is let out of the susceptor behind the substrate and said circumferential 25 walls and by that the substrate and said reactive gases are heated above a temperature level from which the reactive gases start to decompose into substances depositing on the substrate for the growth of a said object, characterized in 30 that an additional flow of gases not participating in said growth is conducted substantially in parallel with said first flow along said circumferential walls past the substrate and out through outlet means (31) for preventing said substances deriving from reactive gases decomposed from moving along said circumferential walls past the substrate and 35 the object grown thereon and keeping at least a wall of said

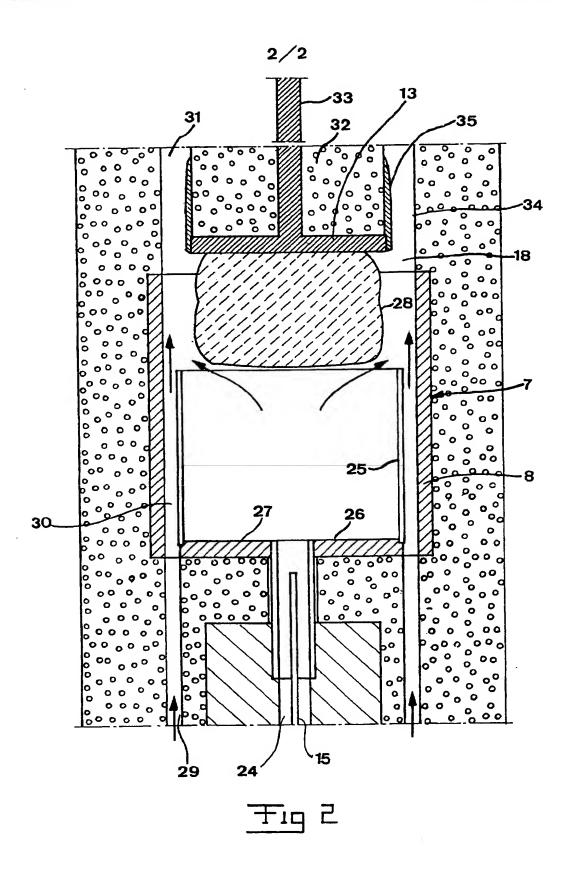
outlet means substantially free from such substances and instead forcing them towards said object, and that the path of said first flow is separated from said circumferential walls and this first flow is guided at a distance therefrom to the region of said object, so that the first flow will be separated from said additional flow to this region.

19. A method according to claim 18, characterized in that said first flow and the flow of additional gases are separated by a physical member ending before the intended location of an upstream end of an object grown as seen in the direction of said flows.

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- 20. A method according to claim 18 or 19, characterized in that an inert gas is fed for forming said additional flow.
  - 21. A method according to any of claims 18-20, characterized in that a gas having a high thermal conductivity is fed for forming said additional flow.
  - 22. A method according to any of claims 18-21, characterized in that He is fed for forming said additional flow.





### INTERNATIONAL SEARCH REPORT

International application No. PCT/SE 97/01613

| A. CLASSIFICATION OF SUBJECT MATTER   |  |                                 |  |  |  |  |  |  |
|---|--|---------------------------------|--|--|--|--|--|--|
| IPC6: C30B 25/14 According to International Patent Classification (IPC) or to both national classification and IPC                        |  |                                 |  |  |  |  |  |  |
| B. FIELDS SEARCHED  | B. FIELDS SEARCHED   |                                 |  |  |  |  |  |  |
| Minimum documentation searched (classification system followed by classification symbols)   |  |                                 |  |  |  |  |  |  |
| IPC6: C30B, C04B  |  |                                 |  |  |  |  |  |  |
| Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched             |  |                                 |  |  |  |  |  |  |
| SE,DK,FI,NO classes as above  |  |                                 |  |  |  |  |  |  |
| Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)              |  |                                 |  |  |  |  |  |  |
|   |  |                                 |  |  |  |  |  |  |
| C. DOCUMENTS CONSIDERED TO BE RELEVANT  | 1  |                                 |  |  |  |  |  |  |
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| Further documents are listed in the continuation of Box   | C. See patent family anne  | x.                              |  |  |  |  |  |  |
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| Date of the actual completion of the international search   | Date of mailing of the international   |                                 |  |  |  |  |  |  |
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| 22 January 1998  Name and mailing address of the ISA/   | Authorized officer   |                                 |  |  |  |  |  |  |
| Swedish Patent Office   |  |                                 |  |  |  |  |  |  |
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# INTERNATIONAL SEARCH REPORT Information on patent family members

07/01/98 PCT/SE

International application No.
PCT/SE 97/01613

|    | Patent document<br>cited in search report |    | Publication date | Patent family member(s) |                                      | Publication date                 |  |
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